

# PATENT SPECIFICATION

**1,199,776**

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NO DRAWINGS

Inventors: RICHARD NORMAN McCARTY and MARTIN CHARLES MUSOLF

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International Classification:—C 08 g 31/26

## COMPLETE SPECIFICATION

### Organosilicon Co-Polymers

We, Dow CORNING CORPORATION, of Midland, Michigan, United States of America, a corporation organised under the laws of the State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

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This invention relates to novel organosilicon compounds which are employed to treat hair on the human head. More particularly, these novel organosilicon compounds are especially suitable for straightening excessively curly hair or curling hair in the event it is straight or after it has become straight and to provide notable improvements in the characteristics and in the appearance of the treated hair.

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In light of the immense interest in hair treatments the cosmetic industry is constantly striving to market new and novel compounds which will provide hair treatments that are a substantial improvement and are superior to those presently known. In the past, organosilicon compounds have been suggested for this use, however they have been generally deficient.

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One inherent disadvantage of organosilicon compounds that have been so employed heretofore is that the compounds have been simply applied to the hair without any chemical bonding. Such being the case, these compounds remain on the hair for only short periods of time because they are readily washed off or otherwise removed from the hair. Also, the prior art organosilicon compounds may improve sheen, body, lustre or humidity resistance, however no specific compound has been able to impart all the above improvements simultaneously.

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It is therefore an object of this invention to introduce and make available novel organosilicon compounds that are not merely applied, but are chemically bonded to the hair so that they will remain on the hair for prolonged and extensive periods of time.

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It is also an object of this invention to impart a more natural look and body to hair which has been subjected to the rigours of waving lotions and to provide hair which will maintain its position through exceptional resistance to humidity and other destructive effects.

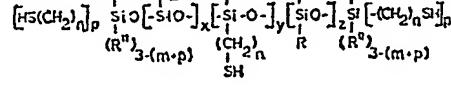
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It is a further object of this invention to provide novel compounds for the treatment of hair whereby the hair retains its original colour, the lustre is improved and made more attractive, the natural characteristics of the hair are retained, and a firmer, longer-lasting curl is obtained.

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It is still a further object of this invention to provide novel compounds that can be employed to straighten excessively curly hair.

This invention provides novel organosilicon co-polymers of the general formula



in which,  
 R is an alkyl radical containing at least 8 carbon atoms,  
 R'' is a lower alkyl radical containing from 1 to 7 carbon atoms,  
 n is an integer of at least 2,  
 p is 0, 1 or 2,  
 m is 0, 1 or 2, the sum of  $m+p$  = not more than 2,  
 x is an integer of at least 1, y and z are 0 or an integer, and when y is 0, p is at least 1, and when z is 0, m is at least 1, and x being larger than the sum of y+z.

For purposes of this invention R can be any alkyl radical comprising at least 8 carbon atoms such as octyl, nonyl, decyl, hendecyl, dodecyl, tridecyl, eicosyl, heneicosyl, octadecyl, octacosyl, nonacosyl, triacontyl, and nonahexacontyl. It has been found that excellent results are achieved when R contains from 8 to 20 carbon atoms.

Also, as herein stated, R'' can be any lower alkyl radical containing from 1 to 7 carbon atoms such as methyl, ethyl, propyl, isopropyl, butyl, isobutyl, amyl, and hexyl, however for practical considerations methyl is to be preferred.

As noted, n has a value of at least 2. Thus the carbon chain to which the —SH group is attached must have two or more carbon atoms. Although the chain can contain many carbon atoms, for purposes of this invention it is preferred that n have a value of either 2 or 3.

It is to be further noted that p is 0, 1 or 2, m is 0, 1 or 2, the sum of  $m+p$  being not greater than 2. Hence, the co-polymer can be endblocked with a silicon atom containing both an R radical and a mercaptoalkyl group or it can be endblocked with two R radicals or two mercaptoalkyl groups. However, in no case can the sum of  $m+p$  exceed two. In addition to being endblocked by a mercaptoalkyl group and/or an R radical, these substituents can also be present along the chain as long as the necessary ratio of dimethyl units to the remaining units is maintained.

According to various preferred embodiments of the invention, R contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, x is 10 and:

- a) the sum of  $m+p$  is 0, and y and z are each 1;
- b) m and p are each 1, and y and z are each 0;
- c) p is 1, m is 0, y is 0, and z is 1;
- d) p is 2, m is 0, y is 0 and z is 1; or
- e) m is 2, p is 0, z is 0 and y is 1.

The novel compounds described herein can be prepared by simply equilibrating the appropriate siloxanes or by co-hydrolysis and condensation of the necessary silanes. The above reaction is generally carried out under nitrogen and in the presence of any well-known base or acid equilibration catalyst, although for best results it is preferred that acid-clay be employed as the catalyst. Temperature is not particularly critical and the reaction will occur at room temperature, however for practical purposes and to facilitate the reaction it is generally conducted by application of heat in the range of from 50° to 100°C. for at least 10 hours. It is to be noted that increasing the temperature decreases the time required to carry the reaction to completion.

It is also of importance to note that the provision of both a mercaptoalkyl group and a higher alkyl radical in the novel co-polymers defined herein results in a multiplicity of advantages when they are applied to the hair. For example, the inclusion of the mercaptoalkyl group means that the compound will be chemically bonded to the hair, whereas the inclusion of the higher alkyl radical imparts a fatty consistency to the hair which affords excellent protection from the ravages that are incumbent due to humidity. In addition, a body and lustre is provided to the hair which has been heretofore unknown.

Depending upon the molecular weight of the co-polymers of this invention, the compound can range from a liquid of low viscosity, representative of a siloxane chain with a few silicon-oxygen linkages or thousands of siloxane units may be present in a molecule to form a solid such as a wax or gum. For ease of handling and application, it is desirable that the co-polymer generally be employed in liquid form, however the nature of the type of hair to be treated will often control the desired physical state of the compound.

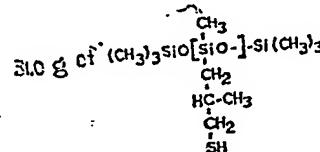
The compounds of this invention are applied to the hair in virtually any form, however the use of the above co-polymers in an aqueous dispersion is most preferred. For example, a very convenient form of application comprises an aqueous dispersion containing about 1 to 10 per cent by weight of the co-polymer. Obviously, if too little

of the co-polymer is employed, a minimum of the desired advantages would be achieved. In contrast, if too great a quantity of the co-polymer is used many advantages will be derived, however, the natural characteristics of the hair may be accordingly destroyed. If the co-polymer is applied in the form of an aqueous dispersion, the use of wetting agents can be employed without deleterious effects, but the use of such agents is not necessary to achieve the objects of this invention.

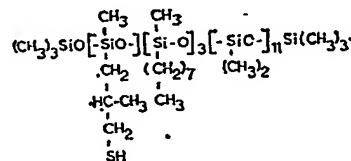
The compounds of this invention applied and reacted on the hair are not harmful to the hair or scalp with which such compounds might come in contact. Water-repellent properties are imparted to the hair strands with the result that they are relatively unaffected by moisture after setting. Thus, treatment of the hair with the co-polymers described herein provides a virtually permanent arrangement of the hair fibres in the presence of high humidity and substantial moisture. The novel compounds of this invention are heat stable at relatively high temperatures and are colourless so that their presence in the hair is undetected and the natural colour of the hair remains throughout. The co-polymers of this invention markedly improve the sheen and appearance of the hair in such a manner as to render the hair more attractive. In addition, sheen-producing agents and the like can be dispensed with, and of extreme importance is the fact that the benefits to be derived are obtained while the hair continues to retain its natural characteristics.

The following examples illustrate the invention.

#### EXAMPLE 1



81.4 g. of mixed cyclopolymers of the unit formula  $[(\text{CH}_3)_2\text{SiO}]$  having an average viscosity of less than 50 cs. at 25°C., 16.2 g. of octylmethylcyclotrisiloxane and 1.2 g. of acid clay were stirred and heated at 60°C under nitrogen for 20 hours. The product was filtered and devolatilised under vacuum (100°C) to give 64.5 g. of a co-polymer of the average formula



#### EXAMPLE 2

Strands of hair were treated in the following manner:

3 min. thioglycollic acid — 3 min. ethanolamine — 3 min. in a 1.0 percent aqueous dispersion of the product of Example 1 — set — 3 min.  $\text{H}_2\text{O}_2$  — uncurled, washed and reset. After the above treatment a long-lasting, tight curl was noted. The curling characteristics of the hair were superior in every respect.

When the strands were then placed in an atmosphere of 80% humidity for approximately 12 hours, the curls retained their original position and the natural characteristics of the hair were maintained.

#### EXAMPLE 3

When the following siloxanes were equilibrated as in Example 1 in the proper proportions, the indicated products were obtained.

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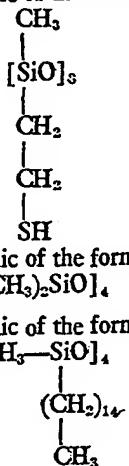
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## SILOXANES

## PRODUCT

I.  
 $(C_2H_5)_3SiOSi(C_2H_5)_3$   
 a cyclic of the formula

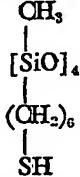


a cyclic of the formula  
 $[(CH_3)_2SiO]_4$

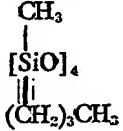
a cyclic of the formula  
 $[(CH_3-SiO]_4$



II.  
 $(CH_3)_3SiOSi(CH_3)_3$   
 a cyclic of the formula



a cyclic of the formula

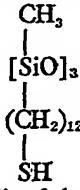


a cyclic of the formula  
 $[(CH_3-Si-O]_4$



III.  
 $(CH_3)_3SiOSi(CH_3)_3$

a cyclic of the formula

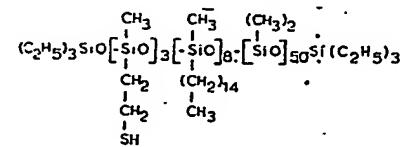


a cyclic of the formula  
 $[(CH_3)_2SiO]_4$

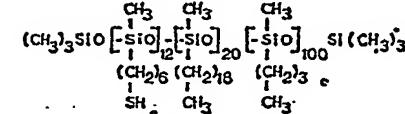
a cyclic of the formula  
 $[(CH_3-Si-O]_3$



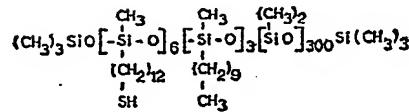
I.



II.



III.



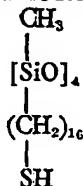
## SILOXANES

## PRODUCT

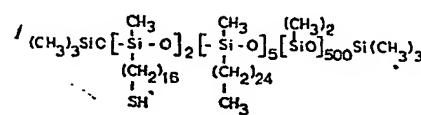
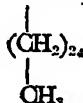
IV.



a cyclic of the formula

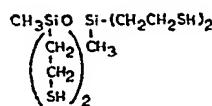


IV.

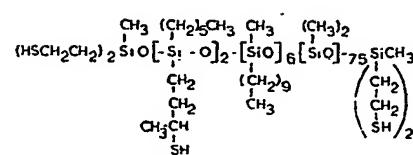
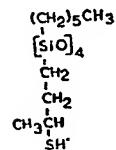
a cyclic of the formula  
[(CH<sub>3</sub>)<sub>2</sub>SiO]<sub>4</sub>a cyclic of the formula  
[CH<sub>3</sub>-Si-O]<sub>5</sub>

V.

V.



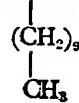
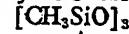
a cyclic of the formula



a cyclic of the formula



a cyclic of the formula

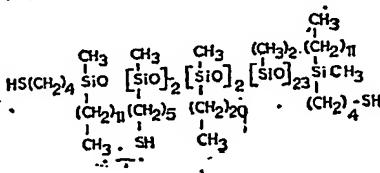
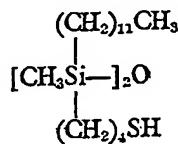


## SILOXANES

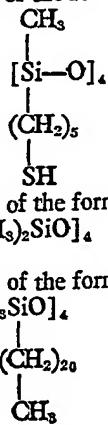
## PRODUCT

VI.

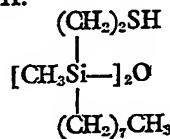
VI.



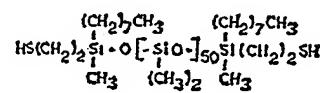
a cyclic of the formula

a cyclic of the formula  
[(CH<sub>3</sub>)<sub>2</sub>SiO]<sub>4</sub>a cyclic of the formula  
[CH<sub>3</sub>SiO]<sub>4</sub>

VII.



VII.

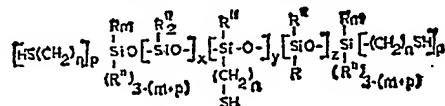


a cyclic of the formula



## WHAT WE CLAIM IS:—

1. An organosilicon co-polymer of the general formula



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in which R is an alkyl radical containing at least 8 carbon atoms,

R'' is a lower alkyl radical containing from 1 to 7 carbon atoms,

n is an integer of at least 2,

p is 0, 1 or 2,

m is 0, 1 or 2,

the sum of m+p being 0 to 2,

x is an integer of at least 1, y and z are each 0 or an integer, and when y is 0, p is at least 1, and when z is 0, m is at least 1, and x being greater than y+z.

10 2. An organosilicon co-polymer as claimed in claim 1 wherein R contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, the sum of m+p is 0, x is 10, and y and z are each 1.

15 3. An organosilicon co-polymer as claimed in claim 1 wherein R contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, m and p are each 1, x is 10, and y and z are each 0.

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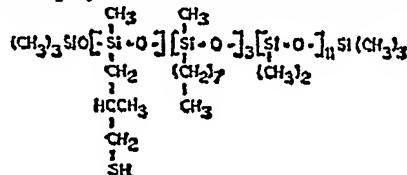
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4. An organosilicon co-polymer as claimed in claim 1 wherein H contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, p is 1, m is 0, y is 0, z is 1, and x is 10.

5 5. An organosilicon co-polymer as claimed in claim 1 wherein R contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, p is 2, m is 0, y is 0, z is 1, and x is 10.

10 6. An organosilicon co-polymer as claimed in claim 1 wherein R contains 8 to 20 carbon atoms, R'' is a methyl radical, n is 2 to 4, m is 2, p is 0, z is 0, y is 1, and x is 10.

10 7. An organosilicon co-polymer as claimed in claim 1 of the formula



**ELKINGTON AND FIFE,**  
Chartered Patent Agents,  
High Holborn House, 52/54, High Holborn, London, W.C.1,  
Agents for the Applicants.

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